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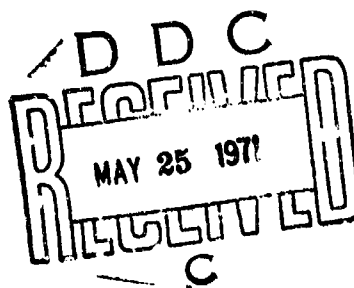
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Port Hueneme, California



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#### IMPREGNATION PLANT IMPROVEMENT TESTS

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#### OBJECT OF PROJECT

To develop chemical warfare decontamination equipment.

#### OBJECT OF SUBPROJECT

To improve chemical handling procedures and equipment for use in converting laundry equipment to a clothing impregnation unit.

#### OBJECT OF REPORT

To present the results of tests to date

#### RESULTS

The mixing time of impregnation chemicals can be greatly reduced by use of a turbine mixing blade and a baffled mixing tank. It is shown that wet clothing can be impregnated for further time-saving in the process.

## SUMMARY

BuDocs passive defense operations require the use of impregnated protective clothing. This clothing is prepared in modified laundry equipment, and chemical preparation equipment to convert a 100 lb combination laundry for impregnation of clothing was designed for development of improved chemical handling. Through a series of tests, a new mixer and tank were developed and procedures were simplified. It is recommended that a single dye compound be prepared for impregnation use. A unit to incorporate the new mixer and procedures will be designed.

## INTRODUCTION

Fulfillment of the BUDOCKS (Bureau of Yards and Docks) passive defense mission requires the preparation of permeable impregnated clothing for chemical warfare protection. Past experience had shown that impregnation of protective clothing could be accomplished in modified portable laundry equipment, and a new combination portable laundry plant was designed for the Bureau by the American Laundry Machinery Corporation, Cincinnati, Ohio, incorporating features which allowed ready conversion to impregnation use. This plant does not include the chemical preparation equipment necessary in impregnation operation, and the primary purpose of the project is the design and testing of the chemical handling equipment for the uses required by the Bureau.

As a starting point for the project tests, NAVCERELAB (U. S. Naval Civil Engineering Research and Evaluation Laboratory) Port Hueneme, California, constructed an impregnation plant using a new laundry unit and chemical handling equipment based on the procedures used in the past. A group of tests was run with the technical cooperation of the NRL (Naval Research Laboratory) to determine the general performance of the plant and normal operating difficulties. A second group was run to determine causes of mixing difficulties and with variations in the dyeing and impregnation procedures to determine potential methods of improving results. As a result of these tests, a chemical handling unit will be designed for use with the combination laundry.

## BASIC LAUNDRY UNIT

The laundry plant consists of three units mounted on steel skids: one 40-in. by 30-in. washer-extractor, a 37-in. by 30-in. drying tumbler, and a steam generator. The plant requires weather protection and a water supply ample for both the steam generator and washer. Electrical power of 208 volt, 60 cycle, 3 phase is required. It can handle loads of 100 lbs, dry weight, of clothing.

The washer-extractor is equipped with an additional drum switch and drive, an impregnate suspension inlet valve, and an impregnate suspension drain valve to allow use as an impregnator. The drum switch is appropriately marked for impregnation operation and changes the extraction speed to 220 rpm for extracting the impregnation slurry. For impregnation operations, the capacity is reduced to 65 to 75 lbs of clothing per load.

## IMPREGNATION CHEMICAL EQUIPMENT

The chemical handling equipment required for preparation of the impregnate suspension must mix and stir the required chemicals in concentrated and diluted suspensions, and pump the dilute suspension in and out of the washer during the impregnation operation. The concentrated suspension is the most difficult to handle, and the normal method has been to use a high speed mixer (1150 rpm) and a circulation gear pump to accomplish the required dispersion of the chemicals to very small particles. The method included in the unit constructed with the new laundry used medium speed mixers (450 rpm) in all tanks. The tanks used were a 150 gal concentrate mixing tank, and two 200 gal dilute suspension tanks. An overall view of the laundry and impregnation chemical tanks is shown in Figure 1. Two gear pumps and appropriate valves and piping to allow transfer of the concentrate and dilute suspension from tank to tank were used.

A scale for weighing chemicals is also included. Figure 2 shows the chemical equipment diagrammatically and Figure 3 shows a photographic view.

## CHEMICALS REQUIRED

The impregnation suspension is designed to deposit the active ingredient, impregnate (CC-2), on the fibers of the clothing throughout the thickness of the fabric. Chlorinated paraffin is used as a binder for the CC-2 and zinc oxide is used to prevent the deterioration of the chlorine strength of the CC-2, and to protect the cloth. To insure penetration and deposition of these materials on the fabric fibers, it is necessary that they be suspended as particles of approximately 4 microns in diameter. Larger particles are filtered out on the surface of the fabric and are unsightly and ineffective. The CC-2 and zinc oxide are normally purchased as a 90 to 10 per cent mixture of "micronized" material called XKCC-3, but the compound is sometimes packed as XKCC-4, a 95-5 mixture of CC-2 and zinc oxide. For impregnation prior to storage, additional zinc oxide to give a 80 - 20, CC-2 to ZnO, ratio is used. The XKCC-3 is composed principally of particles of the proper size, but suspension of the material as individual particles requires vigorous mixing to break down agglomerates of the small particles that normally occur in the packaged material. The chlorinated paraffin is a viscous fluid and must be emulsified to secure the proper particle size.

PVA (Polyvinyl alcohol) -- a dispersing agent, "Daxad" -- a stabilizer, and "Duponal" -- a wetting agent, are used in preparing the suspension to aid in emulsifying and stabilizing the chemicals. Dyes are normally used with the impregnation suspension to give the clothing the proper color.

#### CHEMICAL MIXING

Proper preparation of the suspension is the most critical phase of the impregnation operation and depends on suspending the CC-2, zinc oxide, and chlorinated paraffin as 4 micron particles. Various mixing procedures are used by different military impregnation plants. Some procedures dissolve the PVA separately and prepare the CP emulsion separately; others start with a PVA and XXCC-3 mixture and progressively add the other materials. The mixing equipment available determines to a large extent the procedure most suitable for a given situation. The methods used for the tests were based on an NRL operation instruction<sup>1</sup>

#### TESTS WITH NRL

Since previous Navy impregnation procedures were based on NRL instructions and the chemical equipment had been designed primarily on that basis, a cooperative test arrangement was made with NRL. The tests described under this heading were conducted during July 1956 at Port Hueneme, with the cooperation of Mr. G. H. Fielding of NRL.

#### Concentrate Preparation

Initial step in the test series was preparation of an impregnation suspension concentrate. The equipment used for this initial make-up was the 150 gal round bottomed tank equipped with a medium speed mixer, and the two Gould gear pumps, which are part of the chemical piping and pumping system. PVA and XXCC-4 were mixed dry, then added to water in the mixing tank. The mixture was recirculated through the pumps for 55 minutes. Daxad and Duponal were then added and the mixture was pumped for 25 minutes more. The next morning CP was added to the mixture by using the pump to draw the compound from the drum over a 40 minute period, and the mixture was circulated for an additional 45 minutes. The necessary water was then added. The XXCC-4 powder was added by sprinkling on the surface of the suspension,

but as the addition was being completed, the circulating pump began to stall, despite frequent lubrication with CP. Continuing trouble then occurred with the pumps stalling and with the mixing drum used on the mixer becoming clogged with a pasty mass. The particle size of the XXCC-4 remained too large, being about 40-50 microns. A second batch was then prepared, although difficulty with the gear pumps prevented recirculation of the mix to achieve desired particle-size suspension. Nevertheless, a dilute suspension was prepared from this concentrate, dye was added to the dilute impregnate and a batch of clothing was processed. Several more batches of concentrate were made before the conclusion of testing; none completely satisfactory, although stirring of the concentrate without recirculation seemed to be nearly adequate with at least one of the batches. Unexplained variations in the CC-2 strength of the concentrate occurred, probably because of poor mixing and because the stock chemicals were quite old.

#### Dilute Suspension Preparation

The variations of the concentrate strength carried over into the preparation of the impregnate suspension. Foaming occurred at times but was controllable with Dow-Corning Anti-Foam A. The medium speed mixers were mainly responsible, and low speed paddle mixers will be used for this operation in the new design.

#### Clothing Impregnation

The first batches of clothing were impregnated using a 9 per cent CC-2 suspension, with a resulting deposition of about 14 per cent CC-2 in the clothing fabric. This is considerably higher than the required 10 per cent and consequently experimental runs were made with less concentrated suspensions. Results continued high, however, and the appearance of most of the clothing treated was poor because of mottling and streaking caused by non-uniform deposition of impregnate, possibly because of excess quantities of larger than 4 micron particles in the suspension. Sizing remaining in the material may also have been a cause of the streaking. Some improvement was noted when the more dilute suspensions were used. Experimental variation of the impregnation time seemed to indicate that the exposure time was a more controlling factor than the suspension strength.

#### Extraction of Impregnate

Extraction of the first batch of clothing was carried out at 220 rpm for 3-1/2 minutes and seemed to be inadequate. A second batch was extracted at 660 rpm for periods of 1 to 10 minutes. No definite results were obtained, however. The extraction

method given<sup>1</sup> by NRL was then used. It uses short periods of low speed extraction combined with short periods of tumbling. Slight improvement was noted in the general appearance of the clothing.

#### Drying of Clothing

The NRL instruction manual procedure was used without difficulty and with good results.

#### Process Control Tests

As an effort to simplify the process control tests used in the impregnation scheme, experiments were conducted to check the specific gravity of the concentrate and dilute suspensions. Results indicated that practical difficulties of entrained air and foam interfered with measurements of the specific gravity of the concentrate, but that it was a potentially successful method of checking the CC-2 content of the dilute suspension.

Cloth swatches and tubes were attached to various items of clothing to check on correlation of CC-2 deposits of such samples with the CC-2 deposits on the clothing. Correlation was poor and the test swatches and tubes showed lower strength deposits.

#### Discussion of First Test Series Results

Results throughout this series of tests showed the difficulties of obtaining a smooth job of impregnation. This is not entirely unreasonable since much of the clothing that had been received in an already impregnated condition from a standard impregnation plant showed similar non-uniform deposition of impregnate and dye. The portable laundry proved quite adequate for its part of the function; the difficulties that were observed were largely in the chemical handling process. Although the chemicals obtained from Navy stores seemed likely to have deteriorated beyond normal usability, the later test batches of concentrate made up into approximately normal quality, even without the use of the gear pumps.

#### SECOND TEST SERIES

Because of the poor results of the first tests, it was decided to obtain as much information as possible from operating impregnation plants and to conduct further tests at Fort Huachuca in the hope of establishing more reliable information.



### Mixing Equipment Experiments

Investigation of the fundamentals of mixing for reduction of agglomerated small particles to the ultimate particle size of the compound indicated that a high shear type of mixer was most desirable for this type of operation. Since this is the desired function of the concentrate mixer while handling the XXCC-3 or 4 suspension, a turbine type mixer was selected as yielding the best high shear mixing conditions. This type mixer is also suitable for creating the CP emulsion. A small mixer of this type was constructed and tested with good results. Consequently, a larger mixer with a variable speed drive was built and tested. A satisfactory concentrate suspension was prepared in less than an hour and no difficulty occurred in pumping the concentrate.

One batch of concentrate was made up using a "Premier" dispersator with good results, confirming the previous indication that stirring alone was adequate to bring the particle size down to that of the individual particles. The "milling" action of the gear pumps is apparently unnecessary for preparing the concentrate. More suitable pumps for pumping the concentrate and dilute suspensions are being investigated; an external bearing gear pump has been tried and was adequate.

The proper shape and baffling of the concentrate mixing tank were also logical areas of investigation for improving the mixing process, and a tank with four vertical side baffles was constructed and used with a "Lightnin" mixer. Results were good, but dead areas existed at the base of the baffles. This was remedied in a second tank by leaving vertical slots at the base of the baffles. Subsequent mixing was improved and this type tank was used for the turbine mixer described previously. The tank and mixer are shown in Figures 4 and 5.

### Extraction Experiments

The results of the initial tests had indicated a slight advantage in using the 220 rpm extraction interrupted by short periods of tumbling and further tests were run using this method and others to compare results. Other methods tried were 660 rpm extraction, and the Brooklyn impregnation plant method. The Brooklyn method also uses 220 rpm extraction speeds interrupted with periods of tumbling, but the time intervals are somewhat longer. Results indicated that the NRL manual method was the most satisfactory. The high speed and Brooklyn methods resulted in less uniform color and more heavy deposits of chemicals. Since the operating characteristics of laundry extractors vary, it is probable that a little experimentation of extraction and tumbling times will yield the best results for individual plants.

### Dyeing and Rinsing Experiments

Although the clothing impregnated, according to NRL manual procedures, was satisfactory, the chemical concentrations on the fabric were higher than required, and some excess deposits occurred at seams and folds. In an effort to improve uniformity, the dyeing procedure was varied, with batches being dyed before impregnation, after impregnation, and with the dye solution being used as a base for the dilute suspensions. Pre-dyeing proved valueless, but the post-dyeing operation yielded some improvement in the uniformity of impregnation. Colors were lighter than normal, however, and stronger dye concentrations would be required if this method is used. The addition of concentrate to the dye solution resulted in blotchy results and some indication that the emulsion had broken.

The post-dyeing results led to a substitution of a post-rinse to get the improved uniformity and this proved to be the case. It is questionable whether the improved results justify adding this step to the process, however.

### Heating of Impregnite

A trial run using impregnite heated to 133 degrees resulted in heavier impregnation, indicating that possibility of speeding up the process by using hot impregnite. Since the time required for impregnation is already short compared to other steps in the process, the additional expense of heating the impregnite is not likely to be worthwhile.

### Dyes

It was difficult to obtain uniform results with the dyes used with the impregnation process. Being used in proportion to the CC-2 content, they are deposited roughly in the same relative strength. Since the CC-2 depositions are variable, the dye likewise yields varying colors. In addition, the use of three separate compounds seems an unjustified complication to the process. Since the Army uses a single compound<sup>2</sup>, the use of a single compound seems a worthwhile reduction in the process, even though a lack of color uniformity may continue. The difference is scarcely greater than the variations caused by differences in age of this type of uniform. As there are considerable existing stocks of the three dyes to be used up, it would seem desirable to blend a single compound from the existing material and make subsequent purchases of a single dye of the proper color.

### Wet or Dry Impregnation

A run was made using washed and extracted clothing that was still wet. Impregnation was satisfactory. Success is reported<sup>3</sup> with this method by the Army Chemical Corps.

### Drying Experiments

One test was made in which impregnated suits were drip dried, extracted and air dried, and extracted and dried in the rotary drier to check effects and suitability of these methods. The rotary drier yielded superior drying results. The other methods are suitable for emergency conditions.

### Discussion of Second Test Series Results

The portable laundry machinery proved adequate for impregnation operation, with the exception of a tube failure in the steam generator. This type plant is now under in-service testing as a laundry and the performance of the steam generator on the in-service tests is more likely to be a true indication of any potential tube trouble. The plant used in the impregnation tests was operated intermittently with untreated water, conditions which tend to deteriorate tubes prematurely.

The impregnation procedures recommended in the NRL manual proved to be satisfactory, but the chemical handling equipment designed to convert the laundry to an impregnation plant showed two principal difficulties: the concentrate mixer was inadequate and the pumps were subject to frequent stalling. In addition, the shape of the dilute suspension storage tanks made them too high for convenient operation. The turbine mixer and baffled tank have eliminated troubles from the mixing and pumping standpoints, since they reduce the required time of use of the pumps and insure that the pumps handle the concentrate only after all materials have been reduced to the proper size. The shape of the dilute suspension tanks will be designed to reduce their height. A minor revision of piping will reduce the amount of material used.

### CONCLUSIONS

As a result of these tests and investigations, it is concluded that:

- a. The 100 lb laundry can be converted for clothing impregnation with the chemical handling system shown in Figure 2, and the modified mixing tank and a turbine blade mixer.

b. With the turbine mixer and baffle tank, a batch of concentrate can be prepared in approximately one hour, a considerable time saving over using gear pumps for mixing.

c. Wet clothing can be impregnated, resulting in considerable time saving when the clothing requires laundering before impregnating.

d. Use of a single dye compound is desirable.

#### RECOMMENDATIONS

It is recommended that steps be taken to convert existing stocks of the three separate dyes to a single dye compound and make subsequent purchase of a single dye compound.

#### FUTURE PLANS

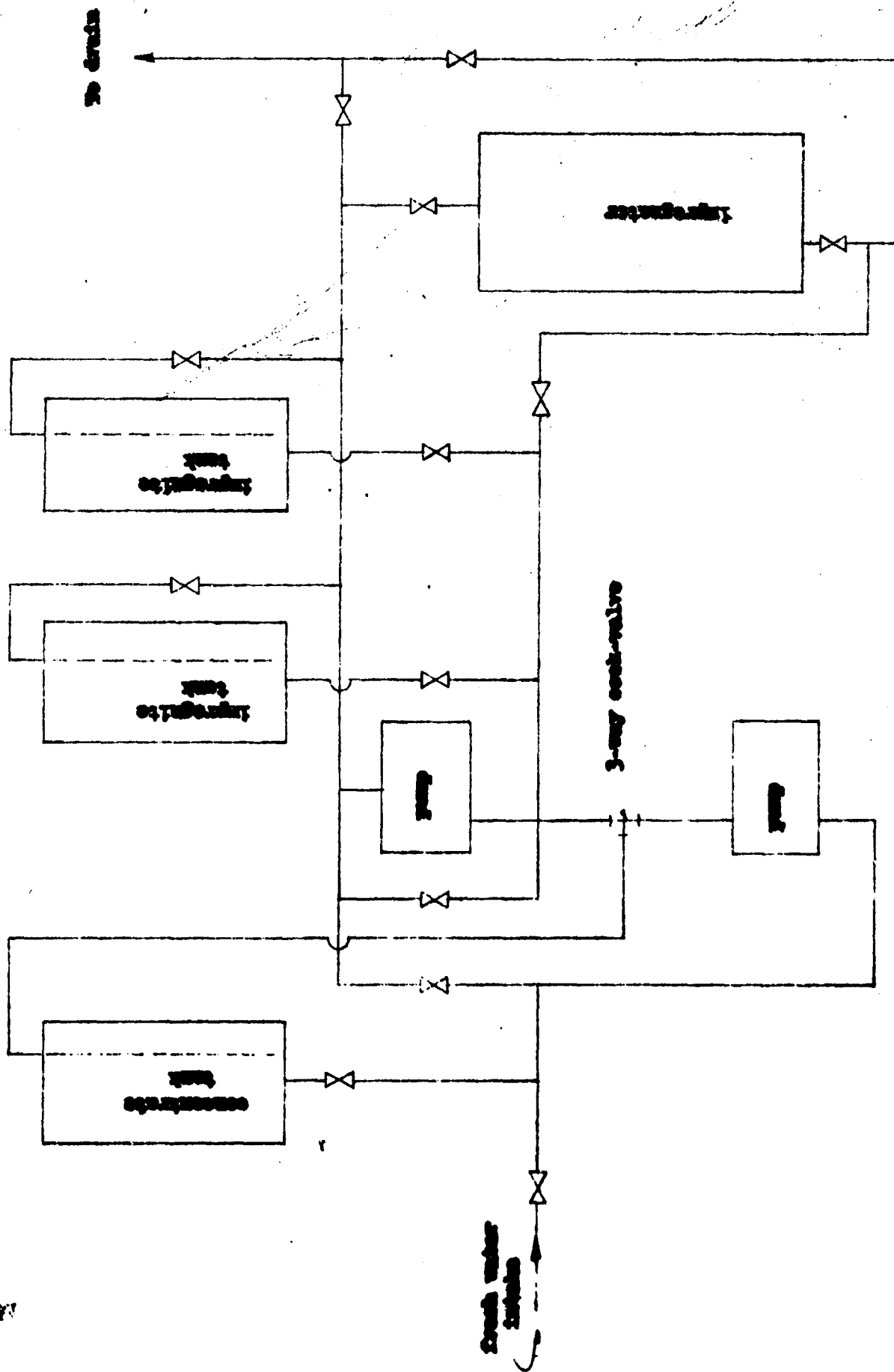
Plans and designs for chemical handling equipment based on the test results will be prepared for use in converting the standard 100 lb laundry to an impregnation plant. Suggested revisions to the NRL operating instructions for operation of the modified plant will be developed.

## REFERENCES

1. Naval Research Laboratory, "Instructions for the Operation of the Navy Portable Clothing Impregnating Plant," A635-B revised by J. M. Davidson, Bureau of Ships.
2. Department of the Army, Technical Manual TM 3-281, "Impregnating Plant, Clothing, M2", February 1954.
3. Chemical Corps, Chemical Warfare Laboratories, "Determination of Method for Impregnation of Wet Clothing," Meredith, John L., 2nd Lt., CRLR 532, Army Chemical Center, Maryland, 6 March 1956.



Figure 1. Chemical Tanks and 100 lb Laundry.



- Schematic -  
 Figure 2. Chemical Regeneration Plant.  
 In scale

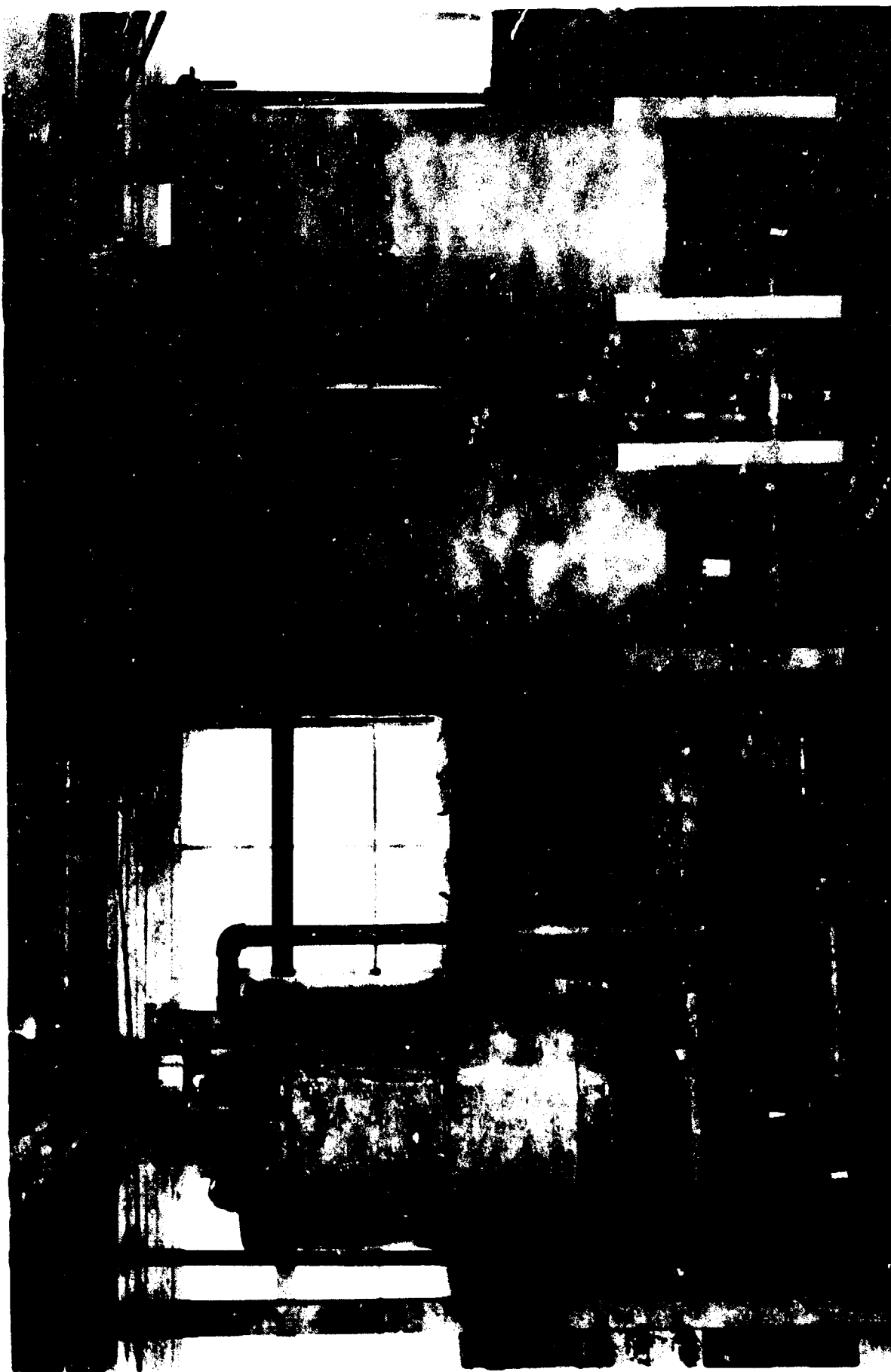


Figure 3. Chemical Mixing Tanks.





**Figure 4. Experimental Mixer with Variable Speed Drive.**

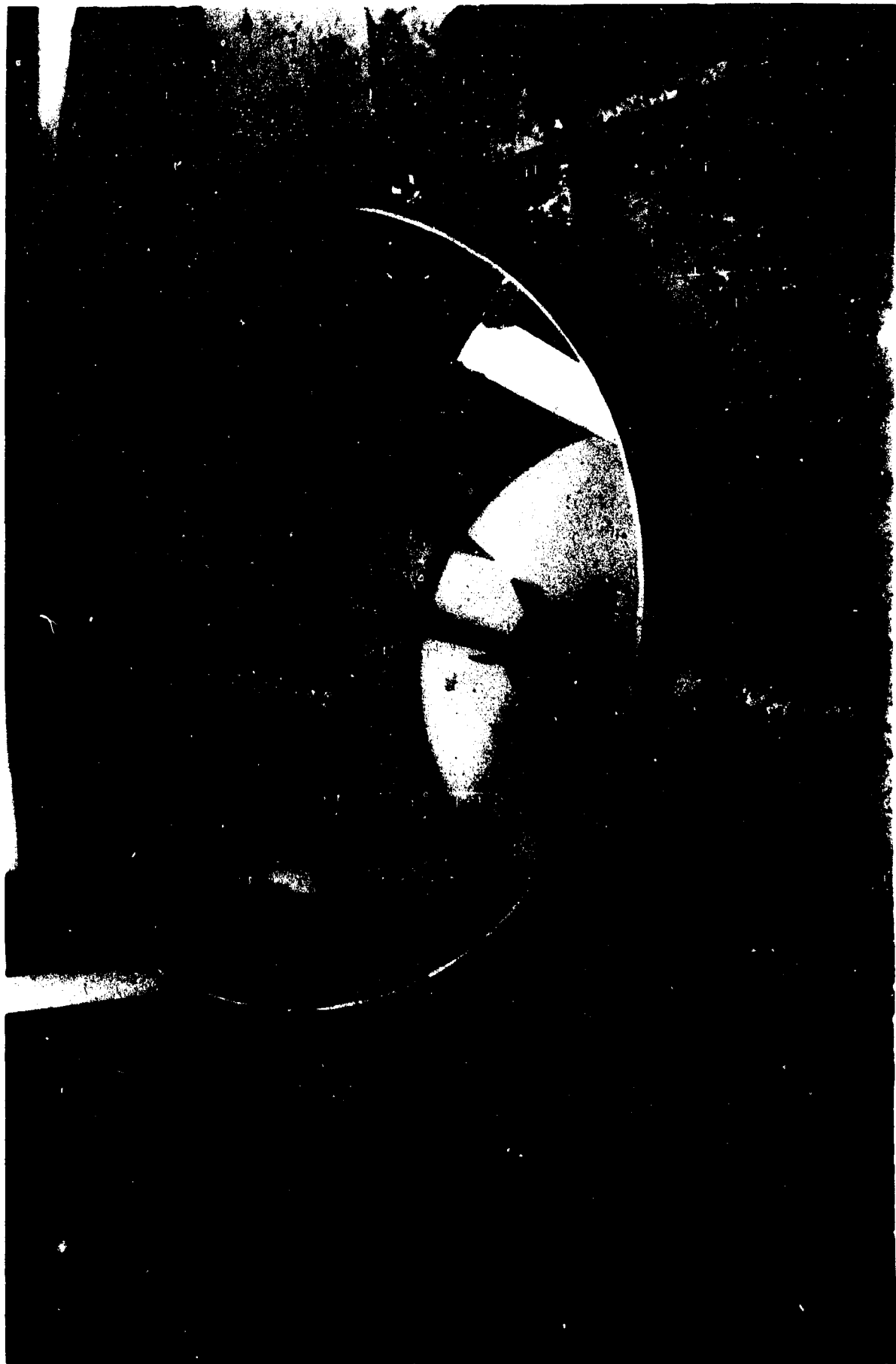


Figure 5. Turbine Blade Mixer in Baffled Tank.